New procedure for the preparation of highly sterically hindered alkenes using a hypervalent iodine reagent

Wiel, Matthijs K.J. ter; Vicario, Javier; Davey, Stephen G.; Meetsma, Auke; Feringa, Bernard

Published in:
ChemInform

Publication date:
2005

```
data_global
_journal_name_full               Org.Biomol.Chem.
_journal_coden_cambridge         0177
_journal_year                    ?
_journal_volume                  ?
_journal_page_first              ?

loop_
_publ_author_name
_publ_author_address
'Ben Feringa'
;
Organic Chemistry
Rijksuniversiteit Groningen
Nijenborgh 4
Groningen
9747 AG
NETHERLANDS
;
'Stephen G. Davey'
;
;
'Javier Vicario'
;
;
'Auke Meetsma'
;
Crystal Structure Center,
Inorganic Solid State Chemistry Laboratory
Chemical Physics,
Materials Science Center,
Groningen University,
Nijenborgh 4,
NL-9747 AG Groningen,
The Netherlands.
;

_publ_contact_author_name        'Ben Feringa'
_publ_contact_author_address
;
Organic Chemistry
Rijksuniversiteit Groningen
Nijenborgh 4
Groningen
9747 AG
NETHERLANDS
;
_publ_contact_author_email       FERINGA@CHEM.RUG.NL

_publ_section_title
;
New procedure for the preparation of highly sterically
hindered alkenes using a hypervalent iodine reagent
;

#=============================================================================

# 1. SUBMISSION DETAILS

_publ_contact_letter             # Include date of submission
;
Date of submission :  2004-09-24  13:39:05
```

```
Consider this CIF submission for deposition of the
X-ray structure of a manuscript is submitted :
Org. Biomol. Chem.

(Our Compound_Identification_Code : CP912)
;
# Publication choise FI, CI or EI for Inorganic
#                    FM, CM or EM for Metal-organic
#                    FO, CO or EO for Organic
_publ_requested_category         ?
_publ_requested_coeditor_name    ?

data_1
_database_code_depnum_ccdc_archive 'CCDC 251068'
#==============================================================================

# 0. AUDIT DETAILS

_audit_creation_date             '2004-06-29  09:19:05'
_audit_creation_method
;
PLATON <TABLE ACC> option
SHELXL97-2 & Manual Editing
;
_audit_update_record
;
?
;

#==============================================================================

# 4. TEXT

_publ_section_synopsis
;
?
;
_publ_section_abstract
;
?
;

# Insert blank lines between paragraphs

_publ_section_comment
; ?
;
_publ_section_exptl_prep
;
?
;
_publ_section_exptl_refinement
;
?
;

# Insert blank lines between references

_publ_section_references
;
Beurskens, P.T., Beurskens, G., Gelder, R. de   Garc\'ia-Granda, S.
Gould, R.O.  Isra\"el, & Smits, J.M.M. (1999).
The DIRDIF99 program system, Technical Report of the Crystallography
Laboratory, University of Nijmegen, The Netherlands.

Bondi, A. (1964). J. Phys. Chem. 68, 441-451.
```

Bruker, (2000). SMART, SAINT, SADABS, XPREP and SHELXTL/NT.
Software Reference Manual Bruker AXS Inc. Madison, Wisconsin, USA.

Flack, H.D. & Bernardinelli, G. (1999). Acta Cryst. A55, 908-915.

Flack, H.D. & Bernardinelli, G. (2000). J. Appl. Cryst. 33, 1143-1148.

International Tables for Crystallography (1983). Vol. A.
Space-group symmetry, edited by T. Hahn. Dordrecht: Reidel.
(Present distributor Kluwer Academic Publishers, Dordrecht).

International Tables for Crystallography (1992). Vol. C.
Edited by A.J.C Wilson, Kluwer Academic Publishers,
Dordrecht, The Netherlands.

Le Page, Y. (1987). J. Appl. Cryst. 20, 264-269.

Le Page, Y. (1988). J. Appl. Cryst. 21, 983-984.

Meetsma, A. (2004). Extended version of the program PLUTO.
Groningen University, The Netherlands. (unpublished).

Sheldrick, G.M. SHELXL97. Program for Crystal Structure
Refinement. University of G\"ottingen, Germany, 1997.

Sheldrick, G.M. SADABS. Version 2. Multi-Scan Absorption Correction Program.
University of G\"ottingen, Germany, 2001

Spek, A.L. (1987). Acta Cryst. C43, 1233-1235.

Spek, A.L. (1988). J. Appl. Cryst. 21, 578-579.

Spek, A.L. (1990). Acta Cryst. A46, C-34.

Spek, A.L. (1994). Am. Crystallogr. Assoc.-Abstracts, 22, 66.
;

_publ_section_figure_captions
;
Fig. 1. Chemical structural diagram (scheme 1) of the title compound

Fig. 2. Perspective PLUTO drawing of the molecule illustrating the
configuration and the adopted numbering scheme.

Fig. 3. Molecular packing viewed down unit cell axes.

Fig. 4. Perspective ORTEP drawing of the title compound.
Displacement ellipsoids for non-H are represented at the 50%
probability level.
The H-atoms are drawn with an arbitrary radius.
;

#==

5. CHEMICAL DATA

_chemical_name_systematic
; ?
;
_chemical_name_common   ?
_chemical_melting_point   ?
_chemical_formula_moiety   'C27 H20 O S'
Ex: 'C12 H16 N2 O6, H2 O' and '(Cd 2+)3, (C6 N6 Cr 3-)2, 2(H2 O)'
_chemical_formula_structural   ?
_chemical_formula_sum   'C27 H20 O S'

```
_chemical_formula_iupac          ?
_chemical_formula_weight         392.52
_chemical_compound_source        'see text'

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
O O 0.0106 0.0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
H H 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
C C 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

#==============================================================================

# 6. CRYSTAL DATA

_symmetry_cell_setting           Orthorhombic
_symmetry_space_group_name_Hall  'P 2ac 2ab'
_symmetry_space_group_name_H-M   'P 21 21 21'
_symmetry_Int_Tables_number      19

loop_
_symmetry_equiv_pos_as_xyz
x,y,z
1/2-x,-y,1/2+z
1/2+x,1/2-y,-z
-x,1/2+y,1/2-z

_cell_length_a                   6.4273(3)
_cell_length_b                   15.6636(8)
_cell_length_c                   18.9920(9)
_cell_angle_alpha                90
_cell_angle_beta                 90
_cell_angle_gamma                90
_cell_volume                     1912.01(16)
_cell_formula_units_Z            4

_cell_measurement_temperature    100(1)
_cell_measurement_reflns_used    6446
_cell_measurement_theta_min      2.60
_cell_measurement_theta_max      29.45
_cell_special_details
;
The final unit cell was obtained from the xyz centroids of
6446 reflections after integration using the SAINT software
package (Bruker, 2000).
;

_exptl_crystal_description       prism
_exptl_crystal_colour            colorless
_exptl_crystal_size_max          0.51
_exptl_crystal_size_mid          0.42
_exptl_crystal_size_min          0.37
_exptl_crystal_size_rad          ?
_exptl_crystal_density_meas      ?
_exptl_crystal_density_diffrn    1.363
_exptl_crystal_density_method    'not measured'
_exptl_crystal_F_000             824
_exptl_absorpt_coefficient_mu    0.186
_exptl_absorpt_correction_type   Multi-Scan
_exptl_absorpt_process_details   '(SADABS, Sheldrick, Bruker, 2000))'
_exptl_absorpt_correction_T_min  0.8686
_exptl_absorpt_correction_T_max  0.9345
```

```
#===============================================================================

# 7. EXPERIMENTAL DATA

_exptl_special_details
; ?
;
_diffrn_ambient_temperature      100(1)
_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type           MoK\a
_diffrn_radiation_source         'fine focus sealed Siemens Mo tube '
_diffrn_radiation_monochromator  'parallel mounted graphite'
_diffrn_radiation_detector
;
CCD area-detector
;
_diffrn_measurement_device_type
;
Bruker Smart Apex
;
_diffrn_measurement_method       'phi and omega scans'
_diffrn_special_details
;
Crystal into the cold nitrogen stream of the low-temparature unit
(KRYOFLEX, (Bruker, 2000)).
;
_diffrn_detector_area_resol_mean '4096x4096 / 62x62 (binned 512)'

_diffrn_standards_number         ?
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time  ?

loop_
_diffrn_standard_refln_index_h
_diffrn_standard_refln_index_k
_diffrn_standard_refln_index_l
? ? ?

# number of measured reflections (redundant set)
_diffrn_reflns_number            17580
_diffrn_reflns_av_R_equivalents  0.0203
_diffrn_reflns_av_sigmaI/netI    0.0195
_diffrn_reflns_limit_h_min       -8
_diffrn_reflns_limit_h_max       8
_diffrn_reflns_limit_k_min       -20
_diffrn_reflns_limit_k_max       20
_diffrn_reflns_limit_l_min       -24
_diffrn_reflns_limit_l_max       25
_diffrn_reflns_theta_min         2.51
_diffrn_reflns_theta_max         28.28
_diffrn_measured_fraction_theta_max 0.992
_diffrn_reflns_theta_full        25.00
_diffrn_measured_fraction_theta_full 0.990

_diffrn_reflns_reduction_process
;
Intensity data were corrected for Lorentz and polarization
effects, decay and absorption and reduced to F~o~^2^
using SAINT (Bruker, 2000) and SADABS (Sheldrick, 2001)
;

# number of unique reflections
_reflns_number_total             4735
_reflns_number_gt                4646
_reflns_threshold_expression     I>2\s(I)
```

```
_computing_data_collection       'SMART, Bruker Version 5.624, 2001'
_computing_cell_refinement       'SAINT, Bruker Version 6.02A, 2000'
_computing_data_reduction        'XPREP, Bruker Version 5.1/NT, 2000'

_computing_structure_solution
;
DIRDIF-99 (Beurskens et al., 1999)
;
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics
;
PLUTO (Meetsma, 2003)
PLATON (Spek, 1994)
;
_computing_publication_material  'PLATON (Spek, 2003)'

#===============================================================================

# 8. REFINEMENT DATA

_refine_special_details
;
Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement.  R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type           full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
'calc w=1/[\s^2^(Fo^2^)+(0.0568P)^2^+0.4536P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     heavy
_atom_sites_solution_secondary   direct
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    refall
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_abs_structure_details
;
Enantiomorph twin refinement
Flack, H.D. & Bernardinelli, G. (1999, 2000)
;
_chemical_absolute_configuration ad

_refine_ls_abs_structure_Flack   0.59(5)
_refine_ls_number_reflns         4735
_refine_ls_number_parameters     343
_refine_ls_number_restraints     0
_refine_ls_number_constraints    ?
_refine_ls_R_factor_all          0.0298
_refine_ls_R_factor_gt           0.0292
_refine_ls_wR_factor_ref         0.0773
_refine_ls_wR_factor_gt          0.0767
_refine_ls_goodness_of_fit_ref   0.956
_refine_ls_restrained_S_all      0.956
_refine_ls_shift/su_max          0.001
_refine_ls_shift/su_mean         0.000

_refine_diff_density_max         0.107
_refine_diff_density_min         -0.076
```

```
_refine_diff_density_rms         0.021

_vrn_publ_code_squeezed_elec     0.0
_vrn_publ_code_void_volume       0.0
_vrn_publ_code_frame_time_sec    5.0
_vrn_publ_code_meas_time_hour    5.5

#===============================================================================

# 9. ATOMIC COORDINATES AND DISPLACEMENT PARAMETERS

loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_thermal_displace_type
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
_atom_site_calc_flag
_atom_site_refinement_flags
S S Uani 1.01488(5) 0.10601(2) 0.17811(1) 1.000 0.0149(1) . .
O O Uani 0.39882(14) 0.10262(6) 0.05637(5) 1.000 0.0182(2) . .
C1 C Uani 0.6617(2) 0.18899(8) 0.11399(6) 1.000 0.0148(3) . .
C2 C Uani 0.7361(2) 0.27092(8) 0.12921(7) 1.000 0.0184(3) . .
C3 C Uani 0.6175(3) 0.34219(8) 0.11171(7) 1.000 0.0241(4) . .
C4 C Uani 0.4246(3) 0.33301(9) 0.08020(7) 1.000 0.0246(4) . .
C5 C Uani 0.3496(2) 0.25235(9) 0.06290(7) 1.000 0.0216(4) . .
C6 C Uani 0.4716(2) 0.18186(8) 0.07885(6) 1.000 0.0161(3) . .
C7 C Uani 0.5532(2) 0.04615(8) 0.03635(6) 1.000 0.0155(3) . .
C8 C Uani 0.5095(2) -0.00980(8) -0.01865(6) 1.000 0.0191(3) . .
C9 C Uani 0.6631(3) -0.06649(9) -0.04078(7) 1.000 0.0214(3) . .
C10 C Uani 0.8579(2) -0.06542(9) -0.00923(7) 1.000 0.0211(4) . .
C11 C Uani 0.8999(2) -0.00920(8) 0.04559(7) 1.000 0.0178(3) . .
C12 C Uani 0.7464(2) 0.04635(7) 0.06996(6) 1.000 0.0141(3) . .
C13 C Uani 0.77101(18) 0.10713(8) 0.13054(6) 1.000 0.0129(3) . .
C14 C Uani 0.75796(19) 0.07039(7) 0.20565(6) 1.000 0.0122(3) . .
C15 C Uani 0.62435(18) 0.11227(8) 0.26148(6) 1.000 0.0129(3) . .
C16 C Uani 0.65047(19) 0.18778(7) 0.30293(6) 1.000 0.0133(3) . .
C17 C Uani 0.8325(2) 0.23958(8) 0.30367(6) 1.000 0.0155(3) . .
C18 C Uani 0.8514(2) 0.30637(8) 0.35029(7) 1.000 0.0196(4) . .
C19 C Uani 0.6894(2) 0.32702(8) 0.39713(7) 1.000 0.0205(3) . .
C20 C Uani 0.5107(2) 0.27990(8) 0.39723(7) 1.000 0.0185(3) . .
C21 C Uani 0.4873(2) 0.20825(7) 0.35157(6) 1.000 0.0149(3) . .
C22 C Uani 0.3096(2) 0.15518(8) 0.35754(7) 1.000 0.0163(3) . .
C23 C Uani 0.29512(19) 0.08062(8) 0.31983(7) 1.000 0.0156(3) . .
C24 C Uani 0.45622(19) 0.05891(8) 0.27261(6) 1.000 0.0142(3) . .
C25 C Uani 0.4742(2) -0.02201(8) 0.22970(6) 1.000 0.0156(3) . .
C26 C Uani 0.7090(2) -0.02511(7) 0.21260(6) 1.000 0.0137(3) . .
C27 C Uani 0.8343(2) -0.06800(8) 0.27138(7) 1.000 0.0179(3) . .
H2 H Uiso 0.86906 0.27760 0.15172 1.00(3) 0.019(3) . .
H3 H Uiso 0.67230 0.39680 0.12375 1.00(3) 0.029(3) . .
H4 H Uiso 0.34215 0.38271 0.06886 1.00(3) 0.034(3) . .
H5 H Uiso 0.21822 0.24236 0.03871 1.00(4) 0.031(3) . .
H8 H Uiso 0.37782 -0.00872 -0.04229 1.00(3) 0.025(3) . .
H9 H Uiso 0.64382 -0.10494 -0.07559 1.00(3) 0.029(3) . .
H10 H Uiso 0.96715 -0.10345 -0.02272 1.00(3) 0.028(3) . .
H11 H Uiso 1.02641 -0.00630 0.06775 1.00(3) 0.015(3) . .
H17 H Uiso 0.94377 0.22688 0.27469 1.00(3) 0.020(3) . .
H18 H Uiso 0.98281 0.33786 0.34987 1.00(4) 0.031(3) . .
H19 H Uiso 0.70360 0.37226 0.42913 1.00(3) 0.018(3) . .
H20 H Uiso 0.40087 0.29101 0.42793 1.00(3) 0.019(3) . .
H21 H Uiso 0.20690 0.16997 0.38856 1.00(3) 0.022(3) . .
H23 H Uiso 0.18280 0.04217 0.32358 1.00(3) 0.017(3) . .
H25 H Uiso 0.42150 -0.07179 0.25294 1.00(3) 0.016(3) . .
```

```
H25' H Uiso 0.39221 -0.01455 0.18619 1.00(3) 0.034(3) . .
H26 H Uiso 0.73484 -0.05223 0.16828 1.00(3) 0.010(3) . .
H27 H Uiso 0.98189 -0.06584 0.25982 1.00(3) 0.024(3) . .
H27' H Uiso 0.81487 -0.04099 0.31663 1.00(3) 0.023(3) . .
H27" H Uiso 0.79148 -0.12821 0.27699 1.00(3) 0.031(3) . .

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
S 0.0115(1) 0.0193(1) 0.0138(1) -0.0005(1) 0.0001(1) -0.0004(1)
O 0.0135(4) 0.0215(4) 0.0195(4) -0.0008(3) -0.0008(3) 0.0006(4)
C1 0.0184(6) 0.0157(5) 0.0102(5) 0.0014(4) 0.0030(4) 0.0014(5)
C2 0.0249(7) 0.0178(6) 0.0126(5) 0.0008(4) 0.0002(5) -0.0017(5)
C3 0.0417(9) 0.0155(6) 0.0150(6) 0.0001(5) 0.0033(6) 0.0018(6)
C4 0.0364(8) 0.0224(6) 0.0149(6) 0.0025(5) 0.0029(5) 0.0123(6)
C5 0.0233(7) 0.0268(7) 0.0148(6) 0.0021(5) 0.0020(5) 0.0076(6)
C6 0.0184(6) 0.0179(5) 0.0120(5) 0.0011(4) 0.0017(5) 0.0017(5)
C7 0.0171(6) 0.0162(5) 0.0133(5) 0.0024(4) 0.0019(4) -0.0010(5)
C8 0.0205(6) 0.0219(6) 0.0150(5) 0.0009(4) -0.0001(5) -0.0058(5)
C9 0.0298(7) 0.0210(6) 0.0133(5) -0.0029(5) 0.0033(5) -0.0048(5)
C10 0.0268(7) 0.0192(6) 0.0172(6) -0.0038(5) 0.0064(5) 0.0024(5)
C11 0.0181(6) 0.0193(6) 0.0159(6) -0.0006(4) 0.0013(5) 0.0012(5)
C12 0.0170(6) 0.0140(5) 0.0113(5) 0.0007(4) 0.0023(4) -0.0002(4)
C13 0.0123(5) 0.0148(5) 0.0117(5) -0.0004(4) -0.0001(4) 0.0002(5)
C14 0.0122(5) 0.0132(5) 0.0112(5) 0.0017(4) 0.0006(4) -0.0005(4)
C15 0.0136(5) 0.0142(5) 0.0108(5) 0.0006(4) -0.0003(4) 0.0023(4)
C16 0.0147(5) 0.0138(5) 0.0114(5) 0.0016(4) -0.0024(4) 0.0019(4)
C17 0.0168(6) 0.0161(5) 0.0137(5) 0.0011(4) 0.0012(5) 0.0004(5)
C18 0.0228(7) 0.0166(6) 0.0194(6) 0.0013(5) -0.0001(5) -0.0033(5)
C19 0.0286(7) 0.0149(5) 0.0180(6) -0.0030(5) -0.0016(5) 0.0012(5)
C20 0.0214(6) 0.0178(5) 0.0163(5) -0.0011(4) 0.0016(5) 0.0042(5)
C21 0.0171(6) 0.0152(5) 0.0124(5) 0.0016(4) -0.0021(5) 0.0028(5)
C22 0.0135(6) 0.0217(6) 0.0138(5) 0.0006(4) 0.0019(5) 0.0033(5)
C23 0.0119(5) 0.0206(5) 0.0142(5) 0.0019(4) -0.0012(4) -0.0006(4)
C24 0.0141(6) 0.0167(5) 0.0118(5) 0.0004(4) -0.0024(4) 0.0005(4)
C25 0.0163(6) 0.0161(5) 0.0145(5) -0.0012(4) 0.0003(5) -0.0017(5)
C26 0.0157(6) 0.0119(5) 0.0134(5) 0.0004(4) 0.0002(4) 0.0005(4)
C27 0.0195(6) 0.0175(6) 0.0166(6) 0.0029(4) -0.0007(5) 0.0019(5)

#===============================================================================

# 10. MOLECULAR GEOMETRY

_geom_special_details
;
Bond distances, angles etc. have been calculated using the
rounded fractional coordinates. All su's are estimated
from the variances of the (full) variance-covariance matrix.
The cell esds are taken into account in the estimation of
distances, angles and torsion angles
;

loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_1
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
S C13 1.8092(12) . . yes
S C14 1.8198(12) . . yes
```

```
O C6 1.3934(16) . . yes
O C7 1.3826(16) . . yes
C1 C2 1.3997(18) . . no
C1 C6 1.3967(18) . . no
C1 C13 1.4955(18) . . no
C2 C3 1.392(2) . . no
C3 C4 1.384(3) . . no
C4 C5 1.392(2) . . no
C5 C6 1.3877(19) . . no
C7 C8 1.3921(17) . . no
C7 C12 1.3962(18) . . no
C8 C9 1.393(2) . . no
C9 C10 1.388(2) . . no
C10 C11 1.3901(19) . . no
C11 C12 1.3945(18) . . no
C12 C13 1.5017(16) . . no
C13 C14 1.5405(16) . . no
C14 C15 1.5140(16) . . no
C14 C26 1.5343(16) . . no
C15 C16 1.4307(17) . . no
C15 C24 1.3824(17) . . no
C16 C17 1.4239(17) . . no
C16 C21 1.4339(17) . . no
C17 C18 1.3759(18) . . no
C18 C19 1.4072(18) . . no
C19 C20 1.3653(18) . . no
C20 C21 1.4262(17) . . no
C21 C22 1.4172(18) . . no
C22 C23 1.3731(18) . . no
C23 C24 1.4114(17) . . no
C24 C25 1.5113(17) . . no
C25 C26 1.5444(18) . . no
C26 C27 1.5317(18) . . no
C2 H2 0.9613 . . no
C3 H3 0.9529 . . no
C4 H4 0.9660 . . no
C5 H5 0.9740 . . no
C8 H8 0.9582 . . no
C9 H9 0.9029 . . no
C10 H10 0.9558 . . no
C11 H11 0.9167 . . no
C17 H17 0.9241 . . no
C18 H18 0.9781 . . no
C19 H19 0.9380 . . no
C20 H20 0.9320 . . no
C22 H21 0.9146 . . no
C23 H23 0.9428 . . no
C25 H25 0.9579 . . no
C25 H25' 0.9870 . . no
C26 H26 0.9574 . . no
C27 H27 0.9743 . . no
C27 H27' 0.9660 . . no
C27 H27" 0.9882 . . no

loop_
_geom_angle_atom_site_label_1
_geom_angle_atom_site_label_2
_geom_angle_atom_site_label_3
_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
C13 S C14 50.24(5) . . . yes
C6 O C7 114.41(10) . . . yes
C2 C1 C6 118.09(11) . . . no
```

```
C2 C1 C13 125.61(11) . . . no
C6 C1 C13 116.29(11) . . . no
C1 C2 C3 119.93(13) . . . no
C2 C3 C4 120.69(13) . . . no
C3 C4 C5 120.44(14) . . . no
C4 C5 C6 118.37(13) . . . no
O C6 C1 120.75(11) . . . yes
O C6 C5 116.88(11) . . . yes
C1 C6 C5 122.34(12) . . . no
O C7 C8 117.67(11) . . . yes
O C7 C12 120.74(10) . . . yes
C8 C7 C12 121.59(11) . . . no
C7 C8 C9 119.00(12) . . . no
C8 C9 C10 120.09(12) . . . no
C9 C10 C11 120.41(12) . . . no
C10 C11 C12 120.43(12) . . . no
C7 C12 C11 118.43(11) . . . no
C7 C12 C13 116.45(11) . . . no
C11 C12 C13 125.12(11) . . . no
S C13 C1 121.35(9) . . . yes
S C13 C12 117.88(9) . . . yes
S C13 C14 65.24(6) . . . yes
C1 C13 C12 109.45(10) . . . no
C1 C13 C14 119.30(10) . . . no
C12 C13 C14 117.83(10) . . . no
S C14 C13 64.53(6) . . . yes
S C14 C15 125.67(8) . . . yes
S C14 C26 120.65(8) . . . yes
C13 C14 C15 121.17(10) . . . no
C13 C14 C26 117.07(10) . . . no
C15 C14 C26 104.23(9) . . . no
C14 C15 C16 132.61(11) . . . no
C14 C15 C24 106.77(10) . . . no
C16 C15 C24 120.49(11) . . . no
C15 C16 C17 124.96(11) . . . no
C15 C16 C21 116.97(10) . . . no
C17 C16 C21 117.85(10) . . . no
C16 C17 C18 120.81(11) . . . no
C17 C18 C19 121.08(12) . . . no
C18 C19 C20 119.94(12) . . . no
C19 C20 C21 120.88(12) . . . no
C16 C21 C20 119.38(11) . . . no
C16 C21 C22 120.65(10) . . . no
C20 C21 C22 119.86(11) . . . no
C21 C22 C23 120.78(12) . . . no
C22 C23 C24 119.12(11) . . . no
C15 C24 C23 121.67(11) . . . no
C15 C24 C25 111.40(10) . . . no
C23 C24 C25 126.93(11) . . . no
C24 C25 C26 102.39(10) . . . no
C14 C26 C25 100.83(9) . . . no
C14 C26 C27 112.49(10) . . . no
C25 C26 C27 111.98(10) . . . no
C1 C2 H2 119.69 . . . no
C3 C2 H2 120.38 . . . no
C2 C3 H3 117.40 . . . no
C4 C3 H3 121.88 . . . no
C3 C4 H4 120.27 . . . no
C5 C4 H4 119.27 . . . no
C4 C5 H5 123.89 . . . no
C6 C5 H5 117.71 . . . no
C7 C8 H8 121.24 . . . no
C9 C8 H8 119.73 . . . no
C8 C9 H9 123.30 . . . no
C10 C9 H9 116.61 . . . no
C9 C10 H10 122.64 . . . no
```

```
C11 C10 H10 116.93 . . . no
C10 C11 H11 123.20 . . . no
C12 C11 H11 116.37 . . . no
C16 C17 H17 120.49 . . . no
C18 C17 H17 118.59 . . . no
C17 C18 H18 117.03 . . . no
C19 C18 H18 121.88 . . . no
C18 C19 H19 120.75 . . . no
C20 C19 H19 119.30 . . . no
C19 C20 H20 122.49 . . . no
C21 C20 H20 116.58 . . . no
C21 C22 H21 118.99 . . . no
C23 C22 H21 120.16 . . . no
C22 C23 H23 123.76 . . . no
C24 C23 H23 117.12 . . . no
C24 C25 H25 114.03 . . . no
C24 C25 H25' 108.14 . . . no
C26 C25 H25 114.63 . . . no
C26 C25 H25' 110.45 . . . no
H25 C25 H25' 107.06 . . . no
C14 C26 H26 108.75 . . . no
C25 C26 H26 111.61 . . . no
C27 C26 H26 110.79 . . . no
C26 C27 H27 109.42 . . . no
C26 C27 H27' 112.85 . . . no
C26 C27 H27" 110.53 . . . no
H27 C27 H27' 108.13 . . . no
H27 C27 H27" 109.18 . . . no
H27' C27 H27" 106.62 . . . no

loop_
_geom_torsion_atom_site_label_1
_geom_torsion_atom_site_label_2
_geom_torsion_atom_site_label_3
_geom_torsion_atom_site_label_4
_geom_torsion
_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
C14 S C13 C1 110.49(11) . . . . no
C14 S C13 C12 -109.73(11) . . . . no
C13 S C14 C15 -111.33(12) . . . . no
C13 S C14 C26 107.67(11) . . . . no
C7 O C6 C1 -32.97(15) . . . . no
C7 O C6 C5 145.29(11) . . . . no
C6 O C7 C8 -145.73(11) . . . . no
C6 O C7 C12 33.61(15) . . . . no
C6 C1 C2 C3 2.39(19) . . . . no
C13 C1 C2 C3 -179.23(12) . . . . no
C2 C1 C6 O 173.92(11) . . . . no
C2 C1 C6 C5 -4.25(18) . . . . no
C13 C1 C6 O -4.61(16) . . . . no
C13 C1 C6 C5 177.22(11) . . . . no
C2 C1 C13 S 3.41(17) . . . . no
C2 C1 C13 C12 -139.34(12) . . . . no
C2 C1 C13 C14 80.67(16) . . . . no
C6 C1 C13 S -178.19(9) . . . . no
C6 C1 C13 C12 39.06(14) . . . . no
C6 C1 C13 C14 -100.93(13) . . . . no
C1 C2 C3 C4 0.9(2) . . . . no
C2 C3 C4 C5 -2.6(2) . . . . no
C3 C4 C5 C6 0.8(2) . . . . no
C4 C5 C6 O -175.58(12) . . . . no
C4 C5 C6 C1 2.66(19) . . . . no
```

O C7 C8 C9 178.95(11) . . . . no
C12 C7 C8 C9 -0.38(19) . . . . no
O C7 C12 C11 -177.08(11) . . . . no
O C7 C12 C13 3.23(16) . . . . no
C8 C7 C12 C11 2.24(18) . . . . no
C8 C7 C12 C13 -177.45(11) . . . . no
C7 C8 C9 C10 -1.5(2) . . . . no
C8 C9 C10 C11 1.4(2) . . . . no
C9 C10 C11 C12 0.5(2) . . . . no
C10 C11 C12 C7 -2.29(18) . . . . no
C10 C11 C12 C13 177.37(12) . . . . no
C7 C12 C13 S 177.25(9) . . . . no
C7 C12 C13 C1 -38.54(14) . . . . no
C7 C12 C13 C14 102.11(13) . . . . no
C11 C12 C13 S -2.42(16) . . . . no
C11 C12 C13 C1 141.79(12) . . . . no
C11 C12 C13 C14 -77.56(15) . . . . no
S C13 C14 C15 117.82(11) . . . . no
S C13 C14 C26 -112.99(10) . . . . no
C1 C13 C14 S -113.46(11) . . . . no
C1 C13 C14 C15 4.36(16) . . . . no
C1 C13 C14 C26 133.54(12) . . . . no
C12 C13 C14 S 109.81(10) . . . . no
C12 C13 C14 C15 -132.37(12) . . . . no
C12 C13 C14 C26 -3.19(16) . . . . no
S C14 C15 C16 3.72(19) . . . . no
S C14 C15 C24 -172.08(9) . . . . no
C13 C14 C15 C16 -75.65(17) . . . . no
C13 C14 C15 C24 108.56(12) . . . . no
C26 C14 C15 C16 149.76(13) . . . . no
C26 C14 C15 C24 -26.04(12) . . . . no
S C14 C26 C25 -174.93(8) . . . . no
S C14 C26 C27 65.62(12) . . . . no
C13 C14 C26 C25 -99.92(11) . . . . no
C13 C14 C26 C27 140.63(11) . . . . no
C15 C14 C26 C25 36.90(11) . . . . no
C15 C14 C26 C27 -82.55(12) . . . . no
C14 C15 C16 C17 -5.7(2) . . . . no
C14 C15 C16 C21 179.79(12) . . . . no
C24 C15 C16 C17 169.59(12) . . . . no
C24 C15 C16 C21 -4.88(17) . . . . no
C14 C15 C24 C23 -177.39(11) . . . . no
C14 C15 C24 C25 3.62(13) . . . . no
C16 C15 C24 C23 6.20(18) . . . . no
C16 C15 C24 C25 -172.79(10) . . . . no
C15 C16 C17 C18 -173.62(12) . . . . no
C21 C16 C17 C18 0.81(17) . . . . no
C15 C16 C21 C20 176.26(11) . . . . no
C15 C16 C21 C22 0.06(17) . . . . no
C17 C16 C21 C20 1.39(17) . . . . no
C17 C16 C21 C22 -174.82(11) . . . . no
C16 C17 C18 C19 -1.87(19) . . . . no
C17 C18 C19 C20 0.7(2) . . . . no
C18 C19 C20 C21 1.61(19) . . . . no
C19 C20 C21 C16 -2.62(18) . . . . no
C19 C20 C21 C22 173.62(12) . . . . no
C16 C21 C22 C23 3.67(19) . . . . no
C20 C21 C22 C23 -172.52(12) . . . . no
C21 C22 C23 C24 -2.55(19) . . . . no
C22 C23 C24 C15 -2.40(19) . . . . no
C22 C23 C24 C25 176.43(12) . . . . no
C15 C24 C25 C26 19.88(13) . . . . no
C23 C24 C25 C26 -159.05(12) . . . . no
C24 C25 C26 C14 -33.94(11) . . . . no
C24 C25 C26 C27 85.87(11) . . . . no

```
loop_
_geom_contact_atom_site_label_1
_geom_contact_atom_site_label_2
_geom_contact_distance
_geom_contact_site_symmetry_1
_geom_contact_site_symmetry_2
_geom_contact_publ_flag
S O 3.3820(10) . 1_655 no
S C6 3.6854(13) . 1_655 no
S C17 3.3820(12) . . no
S C23 3.2630(13) . 1_655 no
S C24 3.4368(12) . 1_655 no
S H2 2.8902 . . no
S H11 2.7374 . . no
S H17 2.6754 . . no
S H23 3.1301 . 1_655 no
S H25' 3.0775 . 1_655 no
S H27 3.1143 . . no
O S 3.3820(10) . 1_455 no
C1 C16 3.5891(16) . . no
C2 C16 3.5895(18) . . no
C2 C17 3.4063(18) . . no
C6 S 3.6854(13) . 1_455 no
C10 C22 3.5981(19) . 2_654 no
C11 C26 3.4100(18) . . no
C16 C1 3.5891(16) . . no
C16 C2 3.5895(18) . . no
C17 S 3.3820(12) . . no
C17 C2 3.4063(18) . . no
C17 C22 3.4925(18) . 1_655 no
C19 C25 3.5355(18) . 4_655 no
C22 C10 3.5981(19) . 2_655 no
C22 C17 3.4925(18) . 1_455 no
C23 S 3.2630(13) . 1_455 no
C24 S 3.4368(12) . 1_455 no
C25 C19 3.5355(18) . 4_645 no
C26 C11 3.4100(18) . . no
C3 H25 2.9131 . 4_655 no
C5 H5 3.0568 . 3_555 no
C6 H5 2.9844 . 3_555 no
C7 H4 2.9468 . 3_555 no
C8 H4 3.0730 . 3_555 no
C8 H19 2.8599 . 4_645 no
C10 H4 3.0795 . 3_555 no
C11 H4 2.9645 . 3_555 no
C11 H26 2.6475 . . no
C12 H26 2.4242 . . no
C12 H4 2.9265 . 3_555 no
C14 H17 3.0255 . . no
C15 H27' 2.8913 . . no
C16 H9 2.9588 . 2_655 no
C17 H21 3.0950 . 1_655 no
C17 H2 2.9560 . . no
C18 H27 3.0867 . 4_755 no
C19 H25' 2.9896 . 4_655 no
C21 H10 2.9121 . 2_655 no
C22 H17 3.0440 . 1_455 no
C22 H10 2.8084 . 2_655 no
C23 H8 3.0596 . 2_555 no
C23 H3 3.0793 . 4_645 no
C24 H27' 2.9088 . . no
C27 H23 2.9963 . 1_655 no
C27 H18 2.9764 . 4_745 no
H2 S 2.8902 . . no
H2 C17 2.9560 . . no
H2 H17 2.5132 . . no
```

```
H3 C23 3.0793 . 4_655 no
H3 H25 2.4678 . 4_655 no
H4 C7 2.9468 . 3_455 no
H4 C8 3.0730 . 3_455 no
H4 C10 3.0795 . 3_455 no
H4 C11 2.9645 . 3_455 no
H4 C12 2.9265 . 3_455 no
H5 C5 3.0568 . 3_455 no
H5 C6 2.9844 . 3_455 no
H8 C23 3.0596 . 2_554 no
H9 C16 2.9588 . 2_654 no
H9 H21 2.5656 . 2_554 no
H10 C21 2.9121 . 2_654 no
H10 C22 2.8084 . 2_654 no
H11 S 2.7374 . . no
H11 H19 2.5755 . 4_745 no
H17 S 2.6754 . . no
H17 C14 3.0255 . . no
H17 C22 3.0440 . 1_655 no
H17 H2 2.5132 . . no
H18 C27 2.9764 . 4_755 no
H18 H26 2.5251 . 4_755 no
H18 H27 2.5820 . 4_755 no
H19 C8 2.8599 . 4_655 no
H19 H11 2.5755 . 4_755 no
H20 H21 2.3891 . . no
H21 C17 3.0950 . 1_455 no
H21 H20 2.3891 . . no
H21 H9 2.5656 . 2_555 no
H23 S 3.1301 . 1_455 no
H23 C27 2.9963 . 1_455 no
H23 H27 2.4487 . 1_455 no
H25 H27" 2.5777 . . no
H25 C3 2.9131 . 4_645 no
H25 H3 2.4678 . 4_645 no
H25' S 3.0775 . 1_455 no
H25' C19 2.9896 . 4_645 no
H26 C11 2.6475 . . no
H26 C12 2.4242 . . no
H26 H18 2.5251 . 4_745 no
H27 S 3.1143 . . no
H27 H23 2.4487 . 1_655 no
H27 C18 3.0867 . 4_745 no
H27 H18 2.5820 . 4_745 no
H27' C15 2.8913 . . no
H27' C24 2.9088 . . no
H27" H25 2.5777 . . no

loop_
_geom_hbond_atom_site_label_D
_geom_hbond_atom_site_label_H
_geom_hbond_atom_site_label_A
_geom_hbond_distance_DH
_geom_hbond_distance_HA
_geom_hbond_distance_DA
_geom_hbond_angle_DHA
_geom_hbond_site_symmetry_A
_geom_hbond_publ_flag
#
#D   H   A   D - H  H...A   D...A    D - H...A  symm(A)
#
C11 H11 S 0.9200 2.7400 3.1839(13) 111.00 . yes
C17 H17 S 0.9200 2.6800 3.3820(12) 134.00 . yes

#===END of Crystallographic Information File
```